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Title: Draft BS EN 13651 Soil improvers and growing media - Extraction of calcium chloride/DTPA (CAT) soluble nutrients.

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Introduction

This draft standard is based on national and international discussions. Your comments on this draft are invited and will assist in the preparation of the consequent standard.

For international standards, comments will be reviewed by the relevant UK national committee before sending the consensus UK vote and comments to the international committee, which will then decide appropriate action. If the international standard is approved, it is usual for the text to be published as a British Standard.

For national standards, comments will be reviewed by the relevant UK national committee and the resulting standards published as a British Standard.

UK Vote

Please indicate whether you consider the UK should submit a negative (with supporting technical reasons) or positive vote on this draft. Please indicate if you are aware of any reason why this draft standard should not be published as a British Standard.

Submission of Comments

- **Annotated drafts are not acceptable and will be rejected.**
- All comments should be submitted online at <https://standardsdevelopment.bsigroup.com/>. You will need to register in order to comment.

Template for comments and secretariat observations

Date: xx/xx/20xx	Document: ISO/DIS xxxx
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1	2	(3)	4	5	(6)	7
MB	Clause No./ Subclause No./Annex (e.g. 3.1)	Paragraph/ Figure/Table/Note	Type of comment	Comment (justification for change) by the MB	Proposed change by the MB	Secretariat observations on each comment submitted
	3.1	Definition 1	ed	Definition is ambiguous and needs clarifying.	Amend to read '...so that the mains connector to which no connection...'	
	6.4	Paragraph 2	te	The use of the UV photometer as an alternative cannot be supported as serious problems have been encountered in its use in the UK.	Delete reference to UV photometer.	

English Version

Soil improvers and growing media - Extraction of calcium
chloride/DTPA (CAT) soluble nutrients

Amendements du sol et supports de culture -
Extraction des éléments nutritifs solubles dans le
chlorure de calcium/DTPA (CAT)

Bodenverbesserungsmittel und Kultursubstrate -
Extraktion von in Calciumchlorid/DTPA (CAT)
löslichen Nährstoffen

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 223.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

This draft European Standard was established by CEN in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Contents

Page

European foreword	3
1 Scope.....	4
2 Normative references.....	4
3 Terms and definitions	4
4 Principle.....	4
5 Reagents	4
5.1 General.....	4
5.2 Reagents.....	4
6 Apparatus	5
6.1 General.....	5
6.2 Apparatus	5
7 Sampling.....	5
8 Test sample	5
9 Procedure	6
9.1 Safety	6
9.2 Extraction of material in solid form	6
9.2.1 Test samples passing through a 20 mm sieve	6
9.2.2 Test samples passing through a 40 mm sieve (≤ 20 % or 20-40 % retained on the sieve) ..	6
9.2.3 Test samples passing through a 40 mm sieve (> 40 % retained on the sieve).....	6
9.3 Extraction of material in pre shaped form	6
9.4 Filtration	6
10 Determination of the extracted nutrients and elements.....	6
11 Expression of results	7
12 Test report.....	7
13 Validation of the method.....	7
13.1 Validation in accordance with ISO 5725-2.....	7
13.2 Performance characteristics.....	7
Annex A (informative) Examples of methods of analysis used for the determination of nutrients and elements	8
Annex B (informative) Performance characteristics of the method	9
Bibliography	26

European foreword

This document (prEN 13651:2025) has been prepared by Technical Committee CEN/TC 223 “Soil improvers and growing media”, the secretariat of which is held by NEN.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 13651:2001.

prEN 13651:2025 includes the following significant technical changes with respect to EN 13651:2001:

- The title has been changed;
- The principle (Clause 4) has been completed;
- The procedure (Clause 8) has been updated with new types of test samples;
- The expression of results (Clause 10) has been completed inserting conversion factors and a calculation on a dry matter basis;
- Methods of analysis (Annex A) have been updated.

This document has been prepared under a standardization request addressed to CEN by the European Commission. The Standing Committee of the EFTA States subsequently approves these requests for its Member States.

1 Scope

This document specifies an extraction method for the routine determination of calcium chloride/DTPA (CAT-method) extractable nutrients and elements in organic soil improver, inorganic soil improver, growing medium, compost and digestate.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CEN/TS 17732:2022, *Soil improvers and growing media - Terminology*

EN 12579:2024, *Soil improvers and growing media - Sampling*

prEN 13040-1:2025, *Soil improvers and growing media — Sample preparation — Part 1: Sample preparation for chemical and physical tests, determination of dry matter content, moisture content and laboratory bulk density*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in CEN/TS 17732:2022 apply.

4 Principle

A sample is extracted with calcium chloride/DTPA at $22\text{ °C} \pm 3\text{ °C}$ in an extraction volume ratio of 1 to 5. The extracted nutrients and elements are determined by various methods as appropriate (see Annex A). If soluble calcium is to be determined, a barium chloride/DTPA solution with the same molar concentration may be used.

5 Reagents

5.1 General

All reagents used shall be of recognized analytical quality and water with a specific conductivity not higher than 0,2 mS/m at 25 °C, free from the elements to be determined.

5.2 Reagents

5.2.1 Dihydrated calcium chloride, $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$.

5.2.2 Diethylenetriaminepentaacetic acid (DTPA), $\text{C}_{14}\text{H}_{23}\text{N}_3\text{O}_{10}$.

5.2.3 Hydrochloric acid, $c(\text{HCl}) = 1:1\text{ vol/vol}$.

5.2.4 Concentrated extracting solution $\text{CaCl}_2/\text{DTPA}$, (CAT), dissolve 14,7 g $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ (5.2.1) and 7,88 g DTPA (5.2.2) in 800 ml hot water (temperature approximately 80 °C) with stirring on a magnetic stirrer in a 1 000 ml beaker. At $75\text{ °C} \pm 10\text{ °C}$ the reagents will dissolve within 2 h. Allow to cool to ambient temperature. Transfer the solution to a 1 000 ml flask and dilute to the mark with water. The solution is stable at room temperature for several weeks. Any precipitation that occurs will disappear with warming and stirring.

5.2.5 Extracting solution $\text{CaCl}_2/\text{DTPA}$, (CAT), dilute the concentrated extracting solution (5.2.4) with water in the proportions one part concentrated solution with nine parts water. The final concentration of the extracting solution should be 0,01 mol/l CaCl_2 and 0,002 mol/l DTPA. The pH of the extracting solution should be adjusted if necessary to be between 2,6 and 2,65 by using HCl (5.2.3).

5.2.6 Nitric acid, $c(\text{HNO}_3) = 15 \text{ mol/l}$; $\rho \approx 1,42 \text{ g/ml}$; not less than 65 % mass/volume.

5.2.7 Nitric acid, $c(\text{HNO}_3) = 0,5 \text{ mol/l}$, dilute 35 ml nitric acid (5.2.6) to one litre with water.

6 Apparatus

6.1 General

NOTE It has been found convenient to carefully clean the equipment (e.g. using nitric acid) and keep separate sets of glassware for the determination of Boron, in order to reduce the possibility of within-laboratory contamination.

All glass apparatus and plastic vessels used in the procedure should be subject to an appropriate preparation procedure in order to keep the risk of contamination as low as possible. It is recommended that all vessels (glass and plastic) are cleansed by carefully immersing in warm nitric acid (5.2.7) for a minimum of 6 h and then rinsed in water.

Rubber stoppers, which may contain traces of metals shall not be used. It is recommended to use plastic or any other stopper free of all substances to be analysed.

Borosilicate glassware shall not be used if boron is to be determined.

6.2 Apparatus

The apparatus consists of the usual laboratory apparatus, and in particular the following:

6.2.1 Plastic bottles or containers, sufficiently large (500 ml to 2 000 ml) with screw cap to accommodate the volume of the sample, extractant and at least 10 % air volume.

6.2.2 Shaking or mixing machine, capable of holding the plastic bottles or containers (6.2.1) and maintaining the sample in suspension without damaging the structure of the sample. The use of a horizontal table shaker is recommended.

6.2.3 Filter paper, cellulose-based ashless types, with a medium pore size of approximately $8 \mu\text{m}$ and diameter of 150 mm.

6.2.4 Analytical balance with an accuracy of 10 mg.

7 Sampling

Sampling is not part of the method specified in this document. EN 12579:2024 shall be followed dealing with soil improvers and growing media. It is important that the laboratory receives a sample that is representative of the product under consideration. The sample should not have been damaged or changed during transport or storage.

8 Test sample

Prepare the laboratory sample in accordance with prEN 13040-1:2025, Clause 9, and determine the laboratory bulk density of the sample in accordance with prEN 13040-1:2025, Annex A.

9 Procedure

9.1 Safety

SAFETY PRECAUTIONS — Care should be taken when handling substances of caustic nature or samples that may contain sharps or is of a dusty nature.

9.2 Extraction of material in solid form

9.2.1 Test samples passing through a 20 mm sieve

Take a weight equivalent to 60 ml of the sample volume (prEN 13040-1:2025, Clause 9.2.5) to the nearest 1 g and transfer to the container (6.2.1). Add 300 ml of extracting solution (5.2.5), secure the cap and shake for 1 h on the shaking machine (6.2.2) at $22\text{ °C} \pm 3\text{ °C}$. Perform the measurement at least 2 times to obtain a mean value.

9.2.2 Test samples passing through a 40 mm sieve ($\leq 20\%$ or $20\text{--}40\%$ retained on the sieve)

Take a weight equivalent to 250 ml of the sample volume (prEN 13040-1:2025, Clause 9.2.3 a) or b)) to the nearest 1 g and transfer to the container (6.2.1). Add 1 250 ml of extracting solution (5.2.5) secure the cap and shake for 1 h on the shaking machine (6.2.2) at $22\text{ °C} \pm 3\text{ °C}$. Perform the measurement at least 2 times to obtain a mean value.

9.2.3 Test samples passing through a 40 mm sieve ($>40\%$ retained on the sieve)

Take a weight equivalent to 300 ml of the sample volume (prEN 13040-1:2025, Clause 9.2.13 c)) to the nearest 1 g and transfer to the container (6.2.1). Add 1 500 ml of extracting solution (5.2.5), secure the cap and shake for 1 h on the shaking machine (6.2.2) at $22\text{ °C} \pm 3\text{ °C}$. Perform the measurement at least 2 times to obtain a mean value.

9.3 Extraction of material in pre shaped form

Take a weight equivalent to 150 ml of the sample volume (prEN 13040-1:2025, Clause 9.5.3) to the nearest 1 g and transfer to the 1 000 ml container (6.2.1). Add 750 ml of extracting solution (5.2.5), secure the cap and shake for 1 h on the shaking machine (6.2.2) at $22\text{ °C} \pm 3\text{ °C}$.

Before filtration pour out the fluid and the pre-shaped sample in a beaker. Squeeze the pre-shaped sample in the beaker as much as possible by applying a pressure to the sample with help of a plastic masher or pestle. Perform the measurement at least 2 times to obtain a mean value.

9.4 Filtration

Filter through filter paper (6.2.3) discarding at least the first 10 ml. In some cases, paper filtration is too slow or even impossible. In such cases, alternative procedures prior to paper filtration (e.g. centrifugation) for obtaining a clear supernatant are acceptable and the technique used shall be reported. The filtered extract is stable for three days in a hermetically closed polyethylene bottle if stored in a refrigerator at $3\text{ °C} \pm 2\text{ °C}$. The filtrate may be stored for longer periods in a deep freezer at about -18 °C .

Before using a solution that has been frozen, the thawed solution should be thoroughly mixed to eliminate gradient separation that occurs on freezing and subsequent thawing.

The measurement of a blank may be used to check possible contaminations of the extracting solution, glassware and filter paper used.

10 Determination of the extracted nutrients and elements

Appropriate methods for the determination of the extracted nutrients and elements shall be used. Examples are provided in Annex A.

11 Expression of results

Subtract the values determined for the reagent blank from those obtained for the samples; results shall be multiplied by 5 (the extraction volume ratio) and expressed in mg/l substrate as received basis.

If results shall be expressed on a dry matter basis (e.g. Nickel), the dry matter content of sample (dm) shall be determined according to prEN 13040-1:2025 (Clause 11) and used in Formula (1):

$$\frac{mg}{kg} = \frac{mg}{l} \times \frac{1}{L_D \times \frac{dm\%}{100}} \times 1000 \quad (1)$$

where

L_D is the laboratory-determined bulk density of fresh sample (in g/l)

$dm\%$ is the dry matter percentage of sample

Phosphorus pentoxide (P_2O_5) shall be calculated by multiplying phosphorus (P) with the factor 2,29. Potassium oxide (K_2O) shall be calculated by multiplying potassium (K) with the factor 1,20. The total extractable nitrogen shall be calculated as the sum of ammonium nitrogen and nitrate nitrogen.

12 Test report

The test report shall contain the following information:

- a) a reference to this document (including its year of publication);
- b) all information necessary for complete identification of the sample;
- c) the results of the determination, expressed as mg/l sample;

NOTE For macronutrients, results can be expressed in whole numbers for microelements with three significant digits.

- d) details of any operations not specified in this document or regarded as optional, as well as any factor which may have affected the results;
- e) the laboratory bulk density;

13 Validation of the method

13.1 Validation in accordance with ISO 5725-2

This standardized reference method was validated in accordance with ISO 5725-2 [1]. All relevant data were reported in an interlaboratory study report, which has been published [15].

13.2 Performance characteristics

The performance characteristics (reproducibility and repeatability) of the method were determined in an interlaboratory study. All data are given in Annex B. The method has been validated for nine different matrices, as listed in Table B.1 to B.17.